



Surface modification of austenitic stainless steel for corrosion resistance in high temperature supercritical-carbon dioxide environment



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ABSTRACT

Surface-modification was applied to austenitic stainless steel 316LN in order to improve the corrosion resistance in high temperature supercritical-carbon dioxide (S-CO₂) environment. The surface-modification methods consisted of deposition of either a single Al layer or a NiAl bi-layer on the surface, followed by inter-diffusion heat treatments. The former resulted in formation of a surface layer composed of a mixture of NiAl and ferrite phases, and the latter resulted in formation of a continuous NiAl surface layer. Surface-modified 316LN showed reduced weight gains than the as-received 316LN alloy after exposure to S-CO₂ at 650 °C (20 MPa) for 500 h. Pre-oxidation in helium at 900 °C before S-CO₂ exposure further improved corrosion resistance by forming inner α-Al₂O₃ layer. On the other hand, the extent of inter-diffusion zone was less for the specimen with a continuous NiAl surface layer before and after S-CO₂ exposure.

1. Introduction

The supercritical-carbon dioxide (S-CO₂) Brayton cycle is a cross-cutting power conversion technology being developed for various energy applications such as next generation nuclear, fossil, and concentrated solar power plants [1–4]. Compared to the conventional steam Rankine cycle and other coolants such as helium (He), the S-CO₂ Brayton cycle is advantageous in terms of efficiency, system footprint, and simplicity of turbomachinery [5].

Past investigations on materials degradation in S-CO₂ environment have mostly focused on evaluation of corrosion and carburization behavior of various candidate materials in S-CO₂. Many researchers reported that ferritic-martensitic steels with less Cr contents such as F91 (9 wt% Cr) exhibit poor corrosion resistance in high temperature S-CO₂ due to formation of thick, duplex Fe- and Cr-rich spinels [6–8]. Austenitic stainless steels show comparatively better corrosion resistances, while those with less Cr contents perform poorly. Anderson et al. exposed a broad range of materials in S-CO₂ at 650 °C and 20 MPa for 3000 h and reported that SS 316 (17 wt% Cr) exhibited significantly higher weight gains compared to other stainless steels with greater Cr

contents [9]. Cao et al. also reported the same for SS 316 and attributed this to chromia (Cr₂O₃) spallation followed by rapid Fe-rich oxide formation, which is indicative of the importance of Cr content for Fe-base alloys [10]. In terms of carburization, it was found that carburization of the Fe-base matrix of Alloy 800HT caused carbide precipitation, which in turn resulted in deterioration of mechanical properties [11].

Ni-base alloys are known to exhibit better corrosion and carburization resistance in high temperature S-CO₂ environment, owing to the formation of a continuous and adherent Cr-rich oxide layer [11–15]. The authors found that 14 wt% Cr was sufficient to form such chromia layer on a Ni-Cr binary alloy in S-CO₂ environment [16]. Moreover, good high temperature creep properties make Ni-base alloys good candidate materials for next generation power plants operating above 700 °C. However, despite these advantages over Fe-base alloys, use of Ni-base alloys would accompany significant increases in cost. Therefore, although Ni-base alloys may be necessary for crucial components such as turbine blades, it may be more practical to use Fe-base alloys for other components that are not expected to be exposed to high stresses.

From this viewpoint, improvement of corrosion resistance of Fe-base alloys in S-CO₂ environment is of practical significance. For this

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Table 1
Chemical compositions of stainless steel 316LN (in wt%).

| Alloy | Fe | Cr | Ni | Mo | Mn | Si | C | N |
|-------|------|------|------|------|------|------|------|------|
| 316LN | Bal. | 18.9 | 13.9 | 2.78 | 1.93 | 0.63 | 0.03 | 0.16 |

purpose, Al additions to the alloys may be considered, as α -alumina (Al_2O_3) is known to provide superior oxidation resistances especially at temperatures above 900 °C. Al may either be alloyed as part of the bulk substrate, or be enriched at the substrate surface. The former method would require detailed assessment and optimization of thermo-mechanical treatments, chemical composition and high temperature properties, all of which would consume significant time. The latter method has the potential benefit that Al content can be increased at the surface for improvement of corrosion resistance while bulk chemical composition and mechanical properties are retained.

While many studies have been performed on improvement of oxidation resistance of Ni-base alloys by means of surface-modification, relatively fewer studies on surface-modification of austenitic stainless steels can be found. Bangaru and Krutenat investigated the application of Al diffusion coatings on various stainless steels, and found that it results in an inter-diffusion layer consisting of NiAl and ferrite phases [17]. Tsai et al. reported similar microstructural changes after aluminate coating on SS 310, and found that the oxidation resistance of aluminized SS 310 was superior especially at 1000 °C in air [18]. Marulanda et al. also found that aluminized austenitic stainless steels showed increased oxidation resistance when exposed to steam at 750 °C for 1000 h [19]. Nonetheless, the effect of such surface-modifications of austenitic stainless steels on corrosion behavior in S-CO₂ and subsequent microstructural evolutions in the surface layer has not been investigated.

In the present work, Al and NiAl diffusion coatings were applied on stainless steel 316LN. In addition, some of the diffusion-coated specimens were pre-oxidized in He at 900 °C for formation of α -alumina prior to exposure to S-CO₂. Then, high temperature S-CO₂ corrosion test was conducted at 650 °C (20 MPa) for 500 h. The corrosion behavior of the surface-modified specimens was discussed in view of the chemical composition and microstructural evolution of the oxide and surface layers.

2. Experimental procedure

2.1. Test materials

The chemical composition of stainless steel 316LN used in this study was analyzed by inductively coupled plasma (ICP) spectroscopy and the results are listed in Table 1. The material was fabricated by electro-discharge machining (EDM) into coupon-type specimens (12 mm in diameter and 1 mm in thickness with a 1.5 mm diameter hole for hanging). The specimens were mechanically ground and polished on both sides by a diamond suspension paste (1 μm) to a mirror-like finish,

Table 2
Working conditions of magnetron sputtering processes.

| Deposited layer | Target | Base pressure | Working pressure (Ar) | DC power (2 gun) | Duration | Coating thickness |
|-----------------|--------------|--------------------------------|-----------------------|------------------|----------|--------------------|
| Al single layer | Al (99.999%) | $\leq 3.0 \times 10^{-6}$ Torr | 0.003 Torr | 50 W | 4 h | ~2.8 μm |
| NiAl bi-layer | Ni (99.99%) | | | | 4 h | ~3.2 μm |
| | Al (99.999%) | | | | 4 h | ~2.8 μm |

then ultra-sonically cleaned in ethanol prior to surface-modification.

2.2. Surface-modification procedure

Magnetron direct current (DC) sputtering method was utilized for coating the specimens. Firstly, the coupon-type specimens were placed on the sputtering rig. Subsequently, the chamber was sealed and evacuated until high vacuum of $< 3.0 \times 10^{-6}$ Torr was attained. Then, magnetron DC sputtering was conducted to form two cases of coatings on the specimens: single layer of Al and a bi-layer of NiAl. The Al layer was approximately 2.8 μm in thickness. For the NiAl bi-layer, Ni layer of about 3.2 μm was first deposited, followed by Al layer deposition of about 2.8 μm on top of the Ni layer. Metal targets of 50.8 mm in diameter were used to deposit the Al and Ni coatings. The thickness of the Al target was 6.35 mm, while it was 1.6 mm for the Ni target. Other working conditions of the magnetron sputtering processes are listed in Table 2.

Then, an inter-diffusion heat treatment (IDHT) was applied to the coated specimens. The IDHT procedure was adopted from a previous work on Alloy 617, and consists of a two-step heat treatment at 600 °C for 0.5 h then at 1000 °C for 0.5 h [20]. The IDHT procedure was conducted in a high vacuum condition ($\leq 3.0 \times 10^{-6}$ Torr) to avoid unintentional oxidation prior to the corrosion test. Following the IDHT process, the coated specimens were subjected to pre-oxidation process at 900 °C for 20 h in high purity He (99.999%) at a flow rate of 150 cm³/min. In a previous work by the authors, pre-oxidation on aluminized Alloy 600 resulted in formation of α -alumina layer, which improved corrosion and carburization resistance in S-CO₂ environment [21]. As result of the above processes, 316LN stainless steels in the as-received and 4 surface-modified conditions were investigated in this study as summarized in Table 3.

2.3. High-temperature S-CO₂ corrosion test

At least two specimens of each condition were subjected to an isothermal corrosion test in S-CO₂ at 650 °C (20 MPa) for 500 h. The specimens were installed in an alumina boat and loaded into the autoclave, after which the autoclave was sealed. Subsequently, research-grade CO₂ (99.999%) was pumped into the autoclave via a CO₂ pump at a rate of 5 mL/min. Meanwhile, the temperature of the autoclave was raised until the test environment reached the target temperature of 650 °C. Then, pressure was continuously increased by the CO₂ pump to 20 MPa and maintained by a back pressure regulator (BPR). Detailed description of the corrosion test facility and procedure can be found in previous works by the authors [11].

For characterization of the microstructural features after surface-modification and oxide products after S-CO₂ exposure, analytical methods such as X-ray diffraction (XRD, Rigaku D/MAX-2500) using X-ray beam at 40 kV voltage and 300 mA current in the range of 20–80° was used. In addition, scanning electron microscopy (SEM, FEI Magellan400) at an accelerating voltage of 10 kV equipped with energy dispersive X-ray spectroscopy (EDS) was conducted. For a detailed

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